

Research Article

# The Importance of the Removal of Helium from Nano-Pd Particles after Solid Fusion

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## Abstract

According to the measuring results of our paper presented at ICCF15 (2009), helium as an important evidence of solid-state fusion has been confirmed clearly by mass analyzer “QMS”. After one solid fusion cycle, the produced helium remained inside the particles. To measure the quantity of helium correctly, the residual helium inside these particles must be completely removed. However, it is not very easy. In this paper, one of the methods to solve the problem of the removal from the nano-Pd particle is discussed.

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*Keywords:* Helium, Nano-Pd particles, Removal, Solid fusion

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## 1. Introduction

Gas loading experiment has been a very important research trend of Low Energy Nuclear Reactions (LENR). We published our nano-Pd studies of excess heat and helium-4 using a new vessel at ICC15 (2009) [1]. The nano-scale Pd particle is attracting more and more researchers to study due to its unique properties [2]. In that paper, throughout the investigation of excess heat and helium-4, we have confirmed the occurrence of “Solid Fusion” by applying nano-Pd powder in  $D_2$  gas loading system. As to measure the quantity of helium correctly, the removal of helium-4 from nano-Pd powder are very important. Therefore, we try to discuss about how to dissolve this problem in this paper.

## 2. Experimental and Discussion

### 2.1. The importance of Pd/D ratio for Solid Fusion

Pd/D ratio is an very important factor for solid fusion. Deuteron diffusing into Palladium expands the lattice. Because of this counter force, most palladium takes-up 0.5–0.7 parts deuteron. Generally very high pressures are needed to get palladium to uptake deuteron beyond PdD0.7. And it is well known that the D/Pd ratio larger than 0.88 is favorable to observe an excess heat during the loading of palladium with deuterium [3,4].

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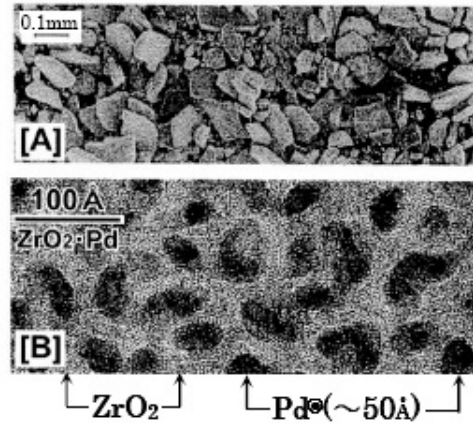


Figure 1. Nano-scale Pd embedded in  $ZrO_2$ .

As shown in Fig. 1, the specimens, discussed in this paper, are nano-scale Pd particles with clusters of about 50 Å in diameter embedded in  $ZrO_2$  matrix [5].

It is well known that nanometer-sized particles display intrinsic different characteristics from those of the corresponding bulk materials due to their unique nanometer-scale structure. The nano-Pd particles work more effectively to absorb large amounts of deuterium and then cause solid fusion attributing to the relative large specific surface area. Therefore, the “solid (state) deuterium”, namely the nuclear fuel, was successfully prepared. The “solid deuterium” is an ultrahigh density deuterium metallic lattice.

We have investigated that D atoms exhibit more stronger effect within host metal clusters and large amounts of D atoms more than 300% against the host atoms can be absorbed in nano-Pd particles, as shown in Fig. 2. Further

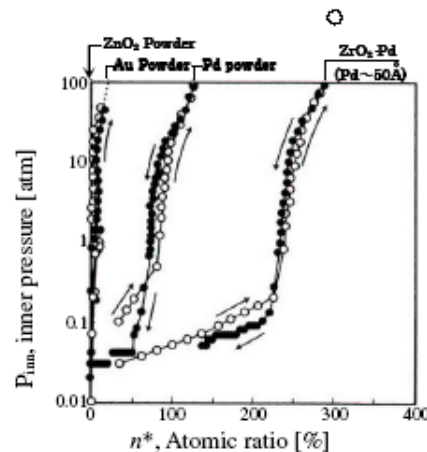
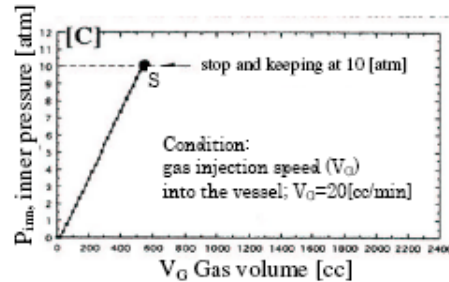


Figure 2.  $D(H)/Pd = 2.9$  at 100 atm.



**Figure 3.** D/H Absorption characteristics of  $ZrO_2$  powder.

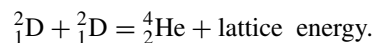
investigation shows that  $ZrO_2$  powder did not absorb  $D_2$  as shown in Fig. 3 ( Fig. 1 (C) of [5]).

Figure 2 shows the relationship between the applied pressure and the atomic ratio of D/Pd. The results demonstrate that  $D_2$  gas is quickly absorbed more than 200 and 250% in the number density of atoms into Pd clusters under the conditions of less than atmospheric pressure and around 3 atm, respectively. Furthermore, an enhancement of absorption up to 290% is gained under such high applied pressure as 100 atm.

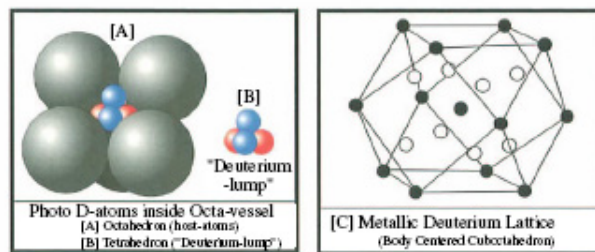
## 2.2. The residual helium-4

As shown in Fig. 4, large amounts of D-atoms absorbed inside nano-Pd are solidified as ultrahigh deuterium-lump (Pycnodeuterium) inside each octahedral space of unit cell of the host Pd lattice [5]. These pycnodeuterium are dispersed to form "metallic deuterium lattice" with body centered cuboctahedron structure, as shown in the right photograph of Fig. 4.

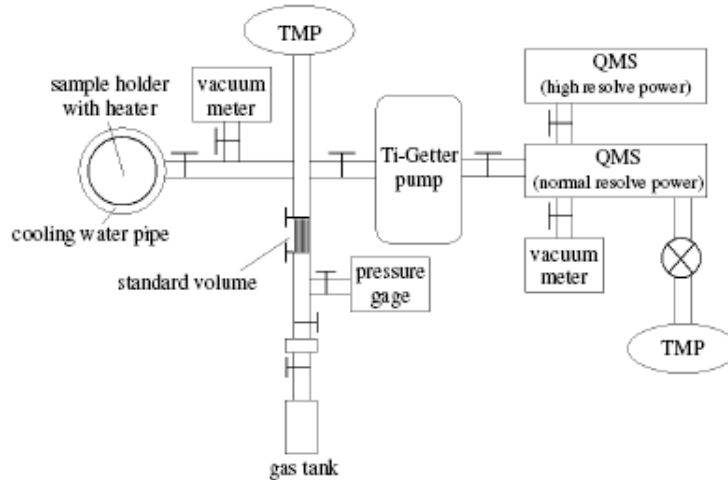
Based on our previous experimental and theoretical analyses results, the high density solid-deuterium fused inside the host Pd lattice (Octa-vessel) by the reaction equation as follows [5],



According to our previous results of solid fusion, helium-4 remained inside both gas and particles after one solid fusion cycle [1]. Therefore, to measure helium-4, the measure process is divided into two parts (two steps), namely the removing of helium-4 from gas and Pd particles, respectively.



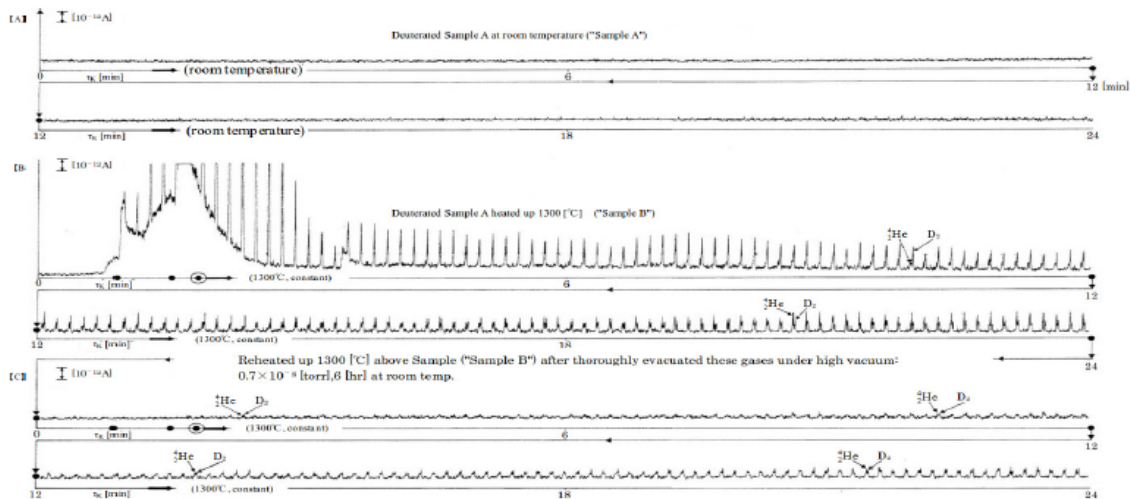
**Figure 4.** Existence position of deuterium in Pd.



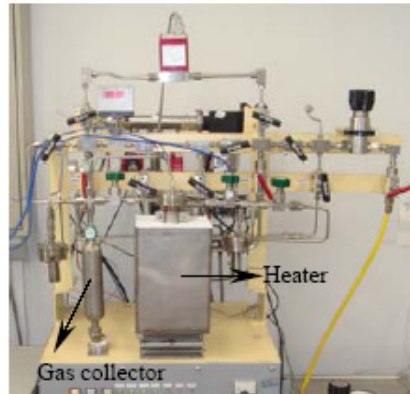
**Figure 5.** Schematic diagram of Mass analysis apparatus — “QMS”.

The exhausting of helium-4 from gas is relatively easy. Applying our measure apparatus, the quadrupole mass spectrometer (QMS) (Fig. 5), the amounts of helium-4 inside reaction gas after one solid fusion cycle can be detected [6].

However, the measurement of helium-4 inside Pd particles is not so easy as gas. According to the results of the previous analysis, it is extremely difficult to completely remove helium-4 from Pd particles. The removing of the residual helium-4 inside Pd particles could not be expected unless by heating up to high temperature and/or dissolving



**Figure 6.** Characteristic of “released gases” ( ${}^3\text{He}$ ;  $\text{D}_2$ ) discharged from “reheated sample”, using “limited QMS”.



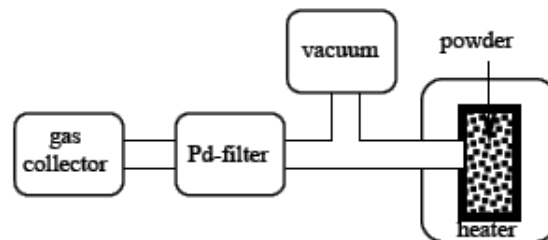
**Figure 7.** The photo of heating facility.

in liquid solution [5].

As stated in the previous report [7], elements released from highly deuterated sample placed in the “closed QMS” with high vacuum ( $\approx 3 \times 10^{-9}$  Torr) are trapped inside the apparatus indefinitely. Helium will continue to exist under the effect of the “getter action” of Ti-Getter pump, shown in Fig. 5. However, the hydrogen series elements and others vanish or diminish to the limit of the Ti-Getter pump’s functional limit. Thus the existence of helium-4 is accurately determined.

In our previous works, we have investigated the releasing action of helium-4 using “limited QMS”(powder inside it can be heated up to over  $1300^\circ$ ) from highly deuterated Pd sample [8], as shown in Fig. 6. In Fig. 6, (A) shows the characteristic of “Sample A” (highly deuterated Pd-black) at room temperature in high vacuum, long period ( $\approx 10^{-8}$  Torr during 6 h. No “couple spectrum” (spectrum of helium-4 and deuteron) can be observed in this case. That is to say that no helium-4 released from sample A. (B) in Fig. 6 shows effect of only “heat” on the above “Sample A” and clear “coupled spectrum” (helium-4 and deuteron) takes place under high temperature and high vacuum ( $\approx 1300^\circ\text{C}$ ,  $\approx 10^{-8}$  Torr). (C) in Fig. 6 shows effect of “reheat” on the above heated sample A (“Sample B”) in (B). Before reheat, all gases released from “Sample B” were thoroughly evacuated under room temperature, high vacuum and long period ( $0.7 \times 10^{-8}$  Torr, 6 h). And when “Sample B” is again heated under the same condition, “couple spectrum” was clearly regenerated.

That is to say, at present, helium-4 inside Pd particles cannot be completely released even heated ramping to  $1300^\circ\text{C}$ .



**Figure 8.** Schematic diagram of the apparatus.

### 2.3. Heating facility

On the basis of those considerations mentioned above, for the production analysis of Solid Fusion, the relationship between the temperature and helium-4 released from nano-Pd particles should be clarified firstly. Therefore, we designed a new heating facility, as shown in Fig. 7.

This heating facility consists of four major parts: heater, gas collector, gas filter and vacuum, as illustrated in Fig. 8. At present, the heat range is designed up to only 1300°C. This temperature is not enough to remove all of helium-4 inside Pd powders. And we are making our efforts to increase the heating ability of this apparatus to fit the temperature requirement.

This heating facility can be used to investigate the characteristic of the extraction of helium-4 from nano-Pd powder after one gas loading cycle. It can also be used to investigate the relationship between the temperature and the release of helium-4 after solid fusion.

Helium-4, due to its unique properties, has been used for leak detection, airships, balloon, scientific uses, and so on. Therefore, as bypass production of solid fusion, helium-4 production can also be expected. The concentrating and collecting of the helium-4 can also be made using this heating facility for use later.

In a word, it is necessary for us to make more efforts on the research of the residual helium-4 using above heating facility.

### 3. Conclusions

To measure helium-4 correctly, after one Solid Fusion cycle, one of the possible methods, heating is discussed in this paper.

Helium-4 inside Pd particles cannot be completely released even by heat ramping to 1300°. It is necessary to make more efforts on the research of the residual helium-4 inside nano-Pd particles.

A new heat facility is designed to investigate the characteristics of helium-4 inside nano-Pd particles after gas loading. And then concentrating and collecting of helium-4 can also be expected using this heat facility.

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